

FTIR Analysis of Apatite Formation on Bioactive Glass Coatings on Ti Alloys

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The objective of our work is to improve the osseointegration of titanium (and its alloys) used in dental and orthopedic applications. To do this, we have been coating these metals with glasses whose compositions are based upon Bioglass®, which is a bioactive glass developed by Hench et al.¹ Bioactive glasses have the property of forming an apatite layer (and therefore can chemically bond to bone) in vivo. Bioglass®, however, will not coat (it cracks) the implant alloys due to large differences in coefficient of thermal expansions. Thus, our group has altered the glass compositions in order to find glasses that will form stable coatings.

After stable coatings have been formed, the level of bioactivity (ability to form apatite) needs to be assessed. To do this, we put samples of coated alloys in simulated body fluid for different lengths of time (from 1 day to 4 months). Once removed, the samples need to be analyzed for apatite formation. Apatite is a calcium phosphate and is the mineral found in bone. There are many forms of apatite including hydroxyapatite, carbonate apatite, and fluoroapatite.

The effects of different variables on the growth of apatite were characterized. Composition effects were determined (i.e. different glasses) with particular emphasis paid to the silica, calcium, and phosphorous contents. The effect of time is also important as to the onset of apatite formation as well as its subsequent growth stages.

We analyzed samples for apatite using fourier transform infrared (FTIR) microscopy at Beamline 1.4.3. Scanning electron microscopy (SEM) with energy dispersive spectroscopy (EDS) was done in conjunction with the FTIR. FTIR at the ALS has several advantages over the other two methods. EDS can only tell us if calcium and phosphorous are present. It is not able to distinguish between various calcium phosphates. FTIR can distinguish apatite by a split P-O bend peak at 610 cm^{-1} and 566 cm^{-1} .² Work at the ALS has shown that apatite has grown on one of our glass coatings (silica content of 57 wt%) after 1 month in simulated body fluid and continues to grow with time while no perceivables changes were detected in the glass coatings with silica content greater than 60 wt% (Fig. 1).

The FTIR at the ALS also has the advantage of having a small spot size. We were able to use this feature to analyze our samples in cross-section. Using this technique along with SEM, we were able to perform line analyses to distinguish the composition and structure of the different layers of our sample: the underlying metal, the glass coating, and the hydroxyapatite layer that forms on the surface (see Fig. 2). The composition and structure of the remaining glass coating did not change after immersion in simulated body fluid an the hydroxyapatite grows on a silica rich layer.

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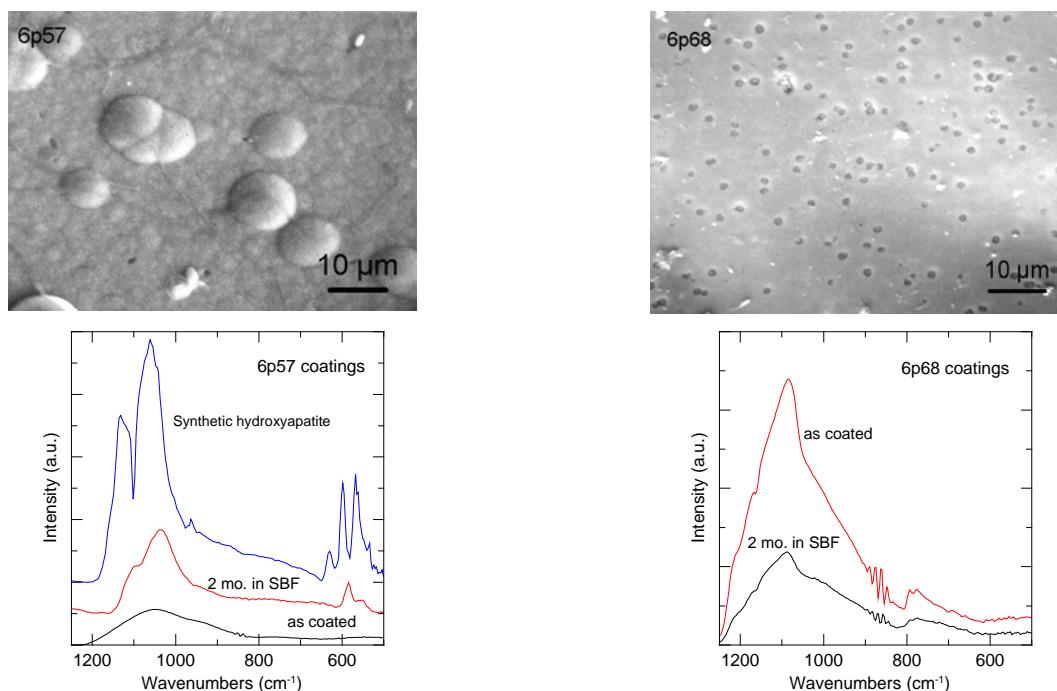


Figure 2 SEM micrographs and corresponding FTIR of the coating surface after immersion on SBF. The silica content of coating 6p68 is 68 wt % and no appreciable change in composition occurs, whereas hydroxyapatite precipitates in coating 6p57 (silica content 57 wt %).

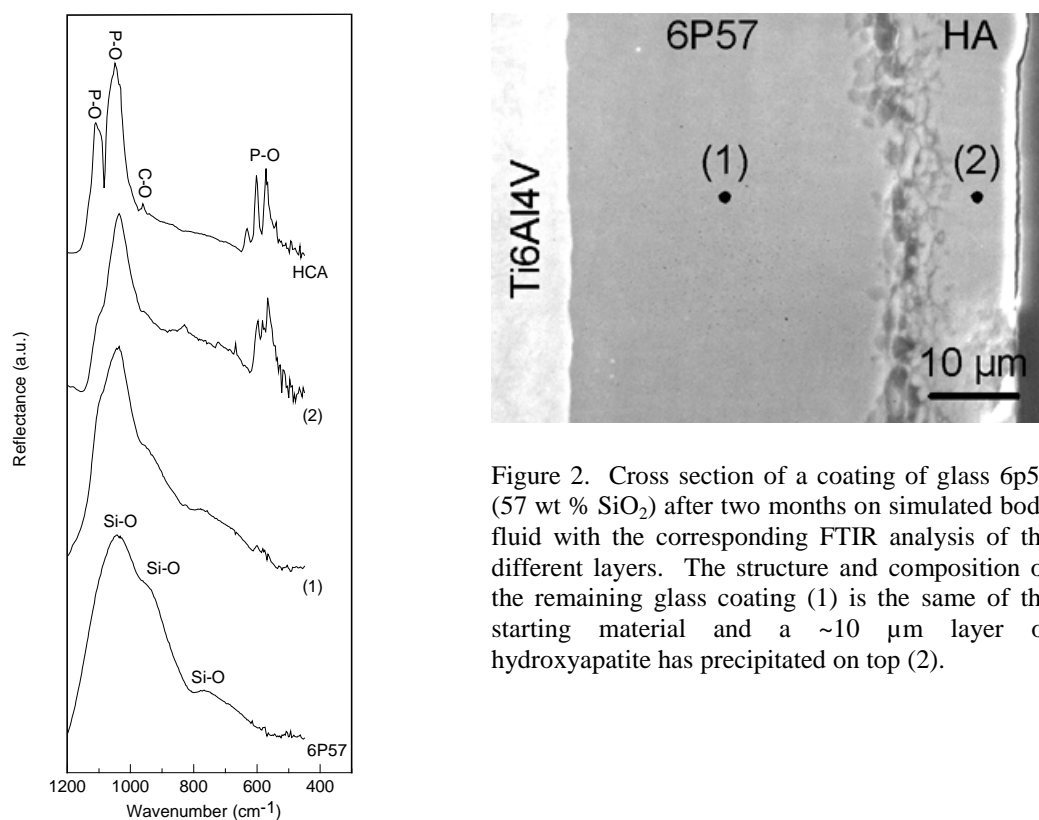


Figure 2. Cross section of a coating of glass 6p57 (57 wt % SiO₂) after two months on simulated body fluid with the corresponding FTIR analysis of the different layers. The structure and composition of the remaining glass coating (1) is the same of the starting material and a ~10 μm layer of hydroxyapatite has precipitated on top (2).